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<p>(21) International Application Number: PCT/GB98/00895 (22) International Filing Date: 24 March 1998 (24.03.98) (30) Priority Data: 97104808 24 March 1997 (24.03.97) RU (71) Applicant (for all designated States except US): STER- ILOX TECHNOLOGIES INTERNATIONAL LIMITED [GB/GB]; The Bindery, Eddington House Estate, Wantage Road, Hungerford, Berkshire RG17 0PL (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): POPOV, Alexey Yurievich [RU/RU]; Solovini pr., 2, 353, Moscow, 117593 (RU). POPOV, Dmitriy Alekseyevich [RU/RU]; Solovini pr., 2, 353, Moscow, 117593 (RU). (74) Agents: VAUGHAN, Christopher, Tammo et al.; Haseltine Lake & Co., Imperial House, 15-19 Kingsway, London WC2B 6UD (GB).</p>		<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p>Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</p>
<p>(54) Title: APPARATUS FOR THE ELECTROCHEMICAL TREATMENT OF WATER AND AQUEOUS SALT SOLUTIONS</p>		
<p>(57) Abstract</p> <p>There is disclosed a method and apparatus for the elec- trolytic treatment of water and aqueous salt solutions. A rela- tively concentrated aqueous salt solution (A) is passed, under pressure, into the working chamber (1) of an electrolytic cell, which cell is divided into a working chamber (1) and an aux- iliary chamber (4) by a permeable membrane (3). A first proportion of the solution (A) passes through the membrane (3) into the auxiliary chamber (4), while a second propor- tion of the solution (A) is passed out of the working chamber (1) and directly recirculated thereto by way of a pipe (12). Gases, such as chlorine, liberated in the working chamber during electrolysis, are dissolved in a water supply (C) so as to generate a supply of water with oxidising properties.</p>		

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APPARATUS FOR THE ELECTROCHEMICAL TREATMENT OF WATER
AND AQUEOUS SALT SOLUTIONS

5 The present invention relates to the
electrochemical treatment of water and aqueous
solutions of salt with the aim of altering the
oxidising and reducing properties of the water or the
aqueous solutions of salt.

10 It is known from Russian patent no. 2038322 to
provide an apparatus for the electrochemical treatment
of water in a midstream diaphragm electrolyser. This
apparatus contains a source of water to be treated, and
water consumption regulators positioned on the lines
for supplying water to the anode and cathode chambers
15 and on the exit from the anode chamber of the
electrolyser. The apparatus also contains a device for
giving a measured supply of reagent to the water being
processed. The shortcoming of this apparatus is the
incomplete use of the reagent being measured, brought
20 about because the reagent, together with the water
being processed, enters both the anode and the cathode
chambers. As a result, when it is necessary to obtain
water with oxidising properties, only the part of the
reagent which enters the anode chamber is usefully
25 used, while the reagent which enters the cathode
chamber with the water is lost. The same phenomenon is
observed when obtaining water with reducing properties.
This results in increased consumption of the reagent,
which makes the electrochemical treatment of the water
30 more expensive and less efficient.

35 It is known from GB 2253860, the disclosure of
which is incorporated into the present application by
reference, to treat water by passing this through an
electrolytic cell having anode and cathode flow
chambers separated by a permeable membrane, one of the
chambers being a working chamber through which water to

be treated passes in an upward direction, and the other being an auxiliary chamber, which is in closed communication with a gas-separating chamber located at a higher level than the electrolytic cell. Water
5 having a higher mineral content than the water to be treated passes upwardly through the auxiliary chamber to the gas-separating chamber and recirculates to the auxiliary chamber by convection and by the shearing forces applied to the water through the rise of bubbles
10 of gas which are generated on the electrode in the auxiliary chamber. The water pressure in the working chamber is higher than that in the auxiliary chamber, and gaseous electrolysis products are vented from the gas-separating chamber by way of a gas-relief valve.

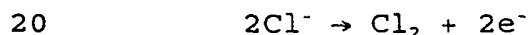
15 This method allows the pH value of the water being treated to be reduced from 7 to around 2 when the anode chamber is used as the working chamber. If instead the cathode chamber is used as the working chamber, the pH value of the water to be treated can be increased to
20 around 12. This known method of electrolytic treatment is applied only to water having a relatively low concentration of dissolved salts and minerals (less than 10gdm^{-3}), and the electricity supplied for the electrolytic treatment of water in the working chamber
25 is only around 200 to 3000Cdm^{-3} . Because the water to be treated has such a low concentration of dissolved salts and minerals, there is consequently a low concentration of useful electrolysis products (such as the chlorate (I) ion ClO^- which is produced when a
30 sodium chloride solution is used in the auxiliary chamber and which acts as a disinfecting agent). In addition, water with a low concentration of salts and minerals tends to have a high ohmic resistance, which means that energy is used inefficiently when performing
35 electrolysis. Furthermore, the small amount of electricity (200 to 3000Cdm^{-3}) applied to the water in

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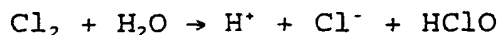
the working chamber is insufficient to ensure the full transformation of the ions of dissolved salts (such as chloride ions Cl^-) into useful electrolysis products (such as chlorate (I) ions ClO^-). The incomplete electrolysis of dissolved salts means that a greater than theoretically necessary amount of salt must initially be dissolved in order to provide a required concentration of electrolysis products. This excess of dissolved salt can mean that the output of the electrolytic cell is overly corrosive, and when used as a disinfectant wash, tends to leave a coating of crystalline salt on surfaces which have been washed.

It is useful to consider the basic chemical reactions which take place in the anode and cathode chambers of the electrolytic cell. If the working chamber contains the anode, then the following reactions take place:

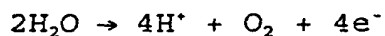
Chloride ions transform into gaseous chlorine at the anode in accordance with the following equation:



Gaseous chlorine dissolves in water and forms hypochlorous acid in accordance with the following equation:

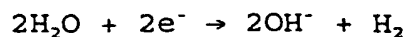


Electrolysis of water also takes place in the anode chamber. The equation is as follows:



As a result of this reaction, gaseous oxygen is liberated and the water becomes saturated with hydrogen ions. Consequently, the pH of the water falls in the anode chamber. The solubility of chlorine in the water reduces as the pH is lowered, and gaseous chlorine is liberated with oxygen.

Electrolysis of water takes place in the cathode chamber. The equation is as follows:



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Consequently gaseous hydrogen is liberated at the cathode, and the concentration of hydroxide ions rises, thereby increasing the water pH in the cathode chamber.

5 It follows from this analysis that the oxidizing ability of water is determined by the concentration of hypochlorous acid, and the reduction ability by the concentration of hydroxide ions. Water which has been under electrolytic treatment according to the method described in GB 2253860 has a low concentration of hypochlorous acid and hydroxide ions due to the low mineralisation of the initial water.

10 One way of estimating the effectiveness of a sterilising solution produced by the electrolytic treatment of a salt solution is to measure the concentration of "free chlorine", by which is understood the concentration of hypochlorous acid in water and the concentration of the chlorate ion (formed by the dissociation of hypochlorous acid).

15 The concentration of free chlorine in water which has been treated in the anode chamber of the electrolytic cell of GB 2253860 does not usually exceed 0.2 to 0.6gdm⁻³, although the solubility of gaseous chlorine in water is much higher (7.3gdm⁻³ at 20°C). It is therefore apparent that water which has been under electrolytic treatment in accordance with the known method has a concentration of free chlorine not more than 3 to 10% of the possible maximum. This low

25 proportion of the chloride ions which are drawn across the permeable membrane from the cathode flow chamber to the anode flow chamber actually reach the anode to be combined so as to form gaseous chlorine. Most of the chloride ions in the anode chamber are carried out of the electrolytic cell with the output of the anode flow chamber before reaching the anode itself.

30 According to a first aspect of the present

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invention, there is provided a method of treating an aqueous salt solution in an electrolytic cell, the cell comprising a working chamber and an auxiliary chamber separated from each other by a permeable membrane, one chamber including an anode and the other a cathode, and each chamber having an input line and an output line, wherein:

i) a relatively concentrated aqueous salt solution is supplied under pressure to the working chamber through its respective input line;

ii) a first proportion of the relatively concentrated solution is filtered under pressure through the permeable membrane into the auxiliary chamber;

iii) an electric current is applied between the cathode and the anode through the aqueous salt solution and the permeable membrane so as to cause electrolysis of the aqueous salt solution;

iv) a second proportion of the aqueous salt solution in the working chamber is output through the output line of the working chamber and directly recirculated to the input line of the working chamber; and

v) gaseous electrolysis products from the working chamber are output through the output line of the working chamber and dissolved in a supply of water so as to generate a product solution.

In preferred embodiments of the present invention, a third proportion of the aqueous salt solution in the working chamber, together with any gaseous electrolysis products from the working chamber, is output through the output line of the working chamber to a gas/liquid separator. The gaseous electrolysis products may then be dissolved in a supply of water, for example mains water, by way of a gas/liquid mixer such as a venturi or the like, and the third proportion of the aqueous

salt solution either discharged or returned to the input line of the working chamber.

Advantageously, a proportion of the aqueous salt solution in the auxiliary chamber is output through the output line of the auxiliary chamber and recirculated, by way of a gas/liquid separation chamber, to the input line of the auxiliary chamber.

According to a second aspect of the present invention, there is provided an apparatus for the electrolytic treatment of an aqueous salt solution, the apparatus comprising an electrolytic cell having a working chamber and an auxiliary chamber separated from each other by a permeable membrane, one chamber including an anode and the other a cathode, and each chamber having an input line and an output line, wherein:

- i) the input line of the working chamber is adapted to receive a supply of aqueous salt solution;
- ii) the working chamber is provided with a recirculation loop in the form of a conduit directly linking the output and the input lines of the working chamber;
- iii) the output line of the working chamber is additionally connected by way of a pressure regulator to a gas/liquid separator provided with a gas output line and a liquid output line, the gas output line being connected to a gas/liquid mixer.

According to a third aspect of the present invention, there is provided an apparatus for the electrochemical treatment of aqueous salt solutions, containing at least one midstream diaphragm electrolyser which contains in turn a working and an auxiliary chamber equipped with separate entry and exit nozzles, characterised in that:

- i) the working chamber is equipped with a closed circulation contour which takes the form of a pipe

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connecting the entry and exit nozzles of the anode chamber;

5 ii) the entry nozzle is connected in its turn by a pipe for supplying aqueous salt solution to the working chamber with a device for increasing the pressure of the aqueous salt solution supplied to the working chamber; and

10 iii) the exit nozzle of the working chamber is connected by a pipe equipped with a pressure regulator to a gas separation tank which is connected in an upper part to a gas and liquid mixer and in a lower part to a discharge pipe.

15 Advantageously, a filter is positioned on the pipe for supplying the aqueous salt solution to the working chamber. This filter helps to remove insoluble impurities which may be present in the supply of aqueous salt solution and which might otherwise clog the permeable membrane.

20 In general, the working chamber contains the anode and the auxiliary chamber contains the cathode, although this arrangement may be reversed in certain applications.

25 For a better understanding of the present invention, and to show how it may be carried into effect, reference shall now be made by way of example to the accompanying Figure, which shows an embodiment of the invention in schematic form.

30 The Figure shows an electrolytic cell comprising an anode chamber 1 which is bounded by an anode 2 and a ceramic semi-permeable diaphragm 3, and a cathode chamber 4 which is bounded by a cathode 5 and the diaphragm 3. The electrolytic cell also has anode and cathode chamber entry nozzles 6 and 7 and exit nozzles 8 and 9 accordingly. The entry nozzle 6 of the anode chamber 1 is connected to a supply pipe 10 which
35 supplies aqueous salt solution (supply A in the Figure)

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and means 11 for increasing the pressure of the aqueous salt solution. The exit nozzle 8 of the anode chamber 1 is connected to the entry nozzle 6 by a pipe 12 which forms the recirculation loop of the anode chamber 1.

5 The exit nozzle 8 of the anode chamber 1 is additionally connected by a pipeline 13 equipped with a pressure regulator 14 to a gas separation tank 15. This tank 15 is connected by a pipe 16 to a gas and liquid mixer 17 positioned on the pipe 18 which is used
10 for supplying water being given oxidising properties to the mixer 17 (shown as supply C in the Figure 1). In the lower part of the gas separation tank 15 is a discharge pipe 19.

A pipe 20 with a stop cock 21 is connected to the
15 exit nozzle 7 of the cathode chamber 4 for supplying auxiliary water or aqueous salt solution to the cathode chamber 4 (shown as supply B in the Figure). The exit nozzle 9 of the cathode chamber 4 is connected by a pipe 22 to a gas separation tank 23, which is connected
20 to the entry nozzle 7 by a pipe 24 which creates a recirculation loop for the cathode chamber 4. The gas separation tank 23 is equipped with a discharge pipe 25 and an outlet 26 for releasing the gaseous products of electrolysis into the atmosphere. A filter 27 may be
25 positioned on the pipe 10 for supplying aqueous salt solution to the anode chamber 1.

In use, aqueous salt solution with a high mineral content (shown as supply A in the Figure), for example
30 a saturated solution of sodium chloride, is supplied to the anode chamber 1 under excess pressure with the help of the device 11. Under the action of this pressure, the aqueous salt solution from the anode chamber 1 penetrates through the ceramic diaphragm 3 and fills the cathode chamber 4. After the anode and cathode
35 chambers 1,4 are filled with aqueous salt solution, an electric current is supplied across the anode 2 and the

cathode 5. Under the action of the electrical current, chloride ions on the surface of the anode 2 are turned into gaseous chlorine, which is partially dissolved in the aqueous salt solution and, partly in the form of gas bubbles together with bubbles of oxygen formed during the electrolysis of the water, is raised into the upper part of the anode chamber 1. The anolyte obtained during the anodic treatment of the aqueous salt solution is drawn along with the gaseous products of electrolysis and leaves the anode chamber 1 by way of the exit nozzle 8. A greater proportion of the anolyte is separated from the gas and is directly returned, by way of pipe 12, to the entry nozzle 6, through which it is returned to the anode chamber 1. In this way, the greater part of the anolyte circulates around a closed contour formed by the anode chamber 1 and the pipe 12 which connects the nozzles 8 and 6. The gaseous products of electrolysis, together with a lesser proportion of the anolyte, enter the gas separation tank 15 by way of pipe 13 and a pressure regulator 14 (which maintains the pressure, necessary for the anolyte to pass through the diaphragm 3 into the cathode chamber 4 from the anode chamber 1). The gaseous products of electrolysis are separated from the anolyte in a tank 15 and enter a gas and liquid mixer 17 (for which a water-jet pump, venturi or similar may be used) by way of a pipe 16. The mixer 17 is positioned on a pipe 18 through which water, for example mains water, enters as supply C. Oxidising properties are given to this water after the gaseous products of electrolysis formed in the anode chamber 1 are dissolved therein. The captured anolyte is discharged from the gas separation tank 15 by way of a pipe 19 and may be returned to the anode chamber 1 for repeated treatment for a fuller use of the remaining dissolved salt. The return of the anolyte from the

tank 15 to the anode chamber 1 is carried out with the help of the device 11 for increasing the water pressure. This device may take the form of a pump connected to an aqueous salt solution supply tank, or a hermetic tank containing such a solution in which excess pressure is created using compressed air, or a tank with containing such a solution which is positioned at a higher level than the electrolytic cell.

In the cathode chamber 4, hydrogen bubbles are formed at the cathode 5 under the action of the electric current. The catholyte, obtained during the processing of aqueous salt solution in the cathode chamber 4, is saturated with hydroxide ions and takes on reducing properties. The hydrogen bubbles, rising into the upper part of the cathode chamber 4, leave by way of the exit nozzle 9 and draw the catholyte behind them; they then pass through a pipe 21 into a gas separation tank 23. In the tank 23, the hydrogen is separated from the catholyte and is passed out into the atmosphere through an outlet 25. The catholyte may be returned from the gas separation tank 23 into the cathode chamber 4 by way of a pipe 22 and the entry nozzle 7. Excess catholyte is discharged from the gas separation tank 23 by way of a pipe 24. The catholyte discharged takes the form of water with reducing properties. Auxiliary aqueous solution (supply B in the Figure) is supplied to the cathode chamber 4 through the entry nozzle 7 by way of a pipe 20. An acid solution intended for removing cathode deposits from the surface of the cathode 5, or mains water supplied to the cathode chamber 4 for cooling the electrolyser in the event of its overheating, may be used as the auxiliary aqueous solution.

A filter 27 may be used for removing insoluble pollutants from the supply A of aqueous salt solution.

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The aqueous salt solution, for example sodium chloride solution, is efficiently processed in the present invention, firstly because the anolyte is subjected to repeated anodic treatment due to the direct recirculation of the aqueous salt solution, and secondly because the anolyte which is carried away from the anode chamber 1 by the gaseous products of electrolysis is separated therefrom in the gas separation tank 15 and returned to the anode chamber 1 for repeated treatment.

A saving of electrical energy is also achieved, because the anode and cathode chambers 1,4 are filled with aqueous salt solutions with a relatively high mineral content and consequent low ohmic resistance. Furthermore, the device 11 for increasing the pressure of the aqueous salt solution supplied to the anode chamber 1 and the pressure regulator 14 positioned on the pipe 13 which connects the exit nozzle 8 of the anode chamber 1 to the gas separation tank 14 helps to ensure that the cathode chamber 4 is supplied through the ceramic diaphragm 3 with aqueous salt solution having a relatively high mineral content.

The provision of a filter 27 on the pipe 10 supplying aqueous salt solution to the anode chamber 1 helps to prevent insoluble impurities in the supply of aqueous salt solution from blocking the pores of the ceramic diaphragm 3.

The performance of an embodiment of the present invention has been experimentally compared with the performance of the device disclosed in GB 2253860. When producing solutions with a single disinfecting activity, the consumption of sodium chloride in the embodiment of the present invention was 7.5 times lower than in the prior art device. The consumption of electrical energy was one half of that of the prior art device.

CLAIMS:

1. A method of treating an aqueous salt solution in an electrolytic cell, the cell comprising a working chamber and an auxiliary chamber separated from each other by a permeable membrane, one chamber including an anode and the other a cathode, and each chamber having an input line and an output line, wherein:

i) a relatively concentrated aqueous salt solution is supplied under pressure to the working chamber through its respective input line;

ii) a first proportion of the relatively concentrated solution is filtered under pressure through the permeable membrane into the auxiliary chamber;

iii) an electric current is applied between the cathode and the anode through the aqueous salt solution and the permeable membrane so as to cause electrolysis of the aqueous salt solution;

iv) a second proportion of the aqueous salt solution in the working chamber is output through the output line of the working chamber and directly recirculated to the input line of the working chamber; and

v) gaseous electrolysis products from the working chamber are output through the output line of the working chamber and dissolved in a supply of water so as to generate a product solution.

2. A method according to claim 1, wherein the working chamber includes the anode and the auxiliary chamber includes the cathode.

3. A method according to claim 1 or 2, wherein the relatively concentrated aqueous salt solution is made up from water and at least one salt selected from the group comprising: sodium chloride, potassium chloride and lithium chloride.

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4. A method according to any one of claims 1 to 3, wherein the relatively concentrated aqueous salt solution is a saturated solution.

5. A method according to any one the preceding claims, wherein a third proportion of the aqueous salt solution in the working chamber, together with any gaseous electrolysis products from the working chamber, is output through the output line of the working chamber to a gas/liquid separator.

6. A method according to claim 5, wherein the third proportion of the aqueous salt solution is returned to the input line of the working chamber.

7. A method according to any one of the preceding claims, wherein a proportion of the aqueous salt solution in the auxiliary chamber is output through the output line of the auxiliary chamber and recirculated, by way of a gas/liquid separation chamber, to the input line of the auxiliary chamber.

8. A method according to any preceding claim, wherein water or a relatively weak aqueous salt solution is supplied to the input line of the auxiliary chamber.

9. An apparatus for the electrolytic treatment of an aqueous salt solution, the apparatus comprising an electrolytic cell having a working chamber and an auxiliary chamber separated from each other by a permeable membrane, one chamber including an anode and the other a cathode, and each chamber having an input line and an output line, wherein:

i) the input line of the working chamber is adapted to receive a supply of aqueous salt solution;

ii) the working chamber is provided with a recirculation loop in the form of a conduit directly linking the output and the input lines of the working chamber;

iii) the output line of the working chamber is

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additionally connected by way of a pressure regulator to a gas/liquid separator provided with a gas output line and a liquid output line, the gas output line being connected to a gas/liquid mixer.

5 10. An apparatus as claimed in claim 9, wherein the working chamber includes the anode and the auxiliary chamber includes the cathode.

10 11. An apparatus as claimed in claim 9 or 10, wherein the permeable membrane is made from a ceramic material.

12. An apparatus as claimed in claim 9, 10 or 11, wherein the permeable membrane is made from zirconium oxide.

15 13. An apparatus as claimed in any one of claims 9 to 12, wherein a gas/liquid separator is connected to the output of the working chamber.

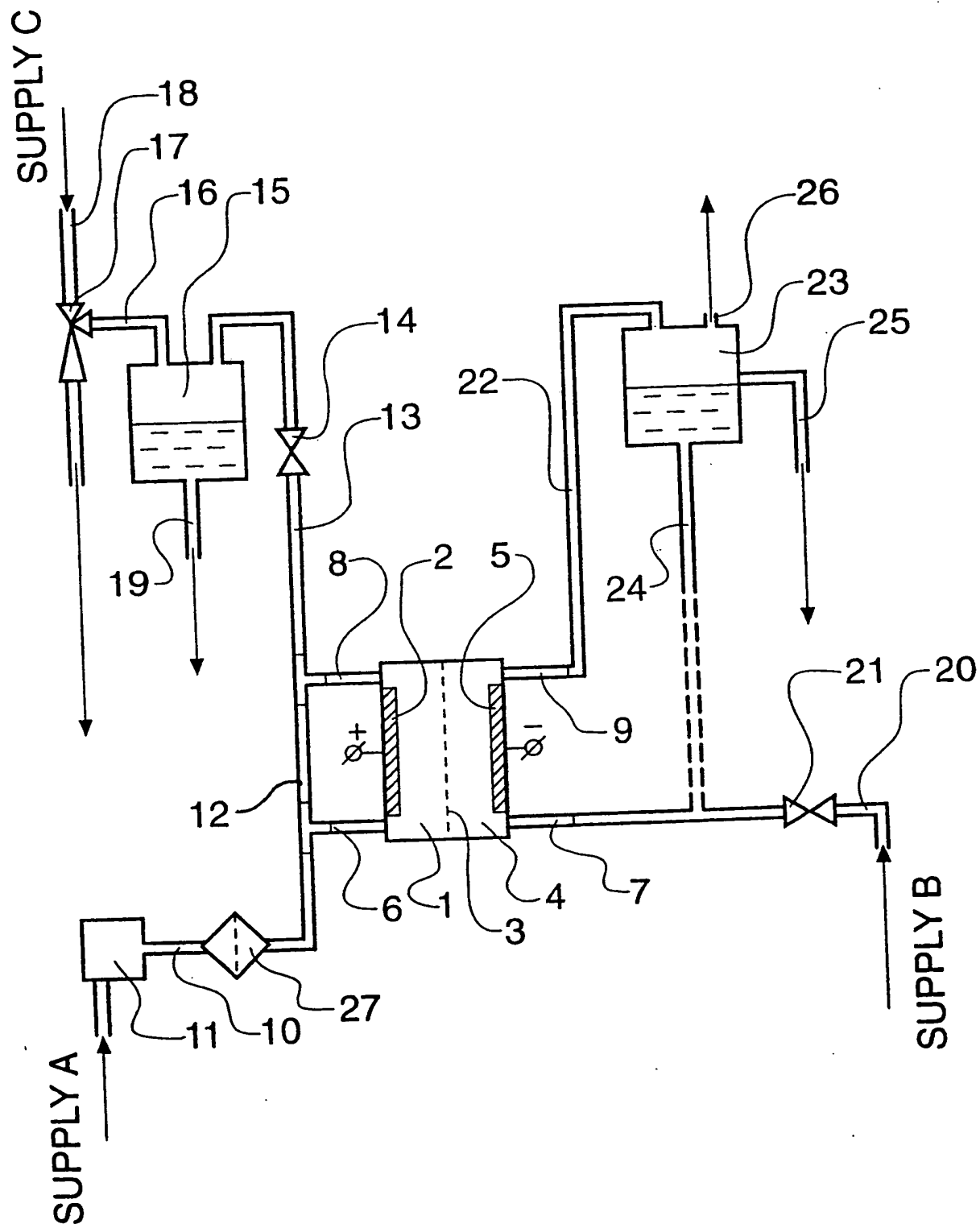
14. An apparatus as claimed in any one of claims 9 to 13, wherein a gas/liquid separator is connected to the output of the auxiliary chamber of the cell.

20 15. An apparatus as claimed in any one of claims 9 to 14, wherein a filter is provided on the input line to the working chamber.

14. A product solution produced by the method of any one of claims 1 to 8.

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 98/00895

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C02F1/461

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C02F C25B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 523 072 A (FALGEN ET AL) 4 June 1996 see column 6, line 20 - line 50; figure 2 see column 5, line 25 - line 28 ----	1-16
A	US 5 443 700 A (HIROSE ET AL) 22 August 1995 see figure 1 ----	
A	US 4 317 704 A (MCINTYRE ET AL) 2 March 1982 see figure 6 ----	
A	US 4 767 511 A (ARAGON) 30 August 1988 see figure 1 ----	
A	GB 2 253 860 A (KIRK AND CHARASHVILI INTERNATI) 23 September 1992 cited in the application -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

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